CLAIMS

1. A continuous process for preparing an organodialkylalkoxysilane of formula (IX):

$$R^1O\text{------}(R^2R^3)Si\text{-----}(CH_2)_3\text{-----}A$$

which consists in continuously contacting an alcohol of formula (VIII): R^1-OH in countercurrent with a silane of formula (VII): Hal---$(R^2R^3)Si-(CH_2)_3$---A, in order to carry out the alcoholysis reaction of said silane according to the following reaction:

$$\underset{(VII)}{Hal\text{—}(R^2R^3)Si\text{-}(CH_2)_3\text{—}A} + \underset{(VIII)}{R^1\text{-}OH} \longrightarrow \underset{(IX)}{R^1O\text{——}(R^2R^3)Si\text{--}(CH_2)_3\text{—}A} + H\text{-}Hal$$

the operation being carried out with stripping of the product of formula H-Hal formed and recovery of the organodialkylalkoxysilane formed in the reactor, in which formulae

- the symbol Hal represents a halogen atom selected from chlorine, bromine and iodine atoms, the chlorine atom being preferred,
- the symbols R^1, which are identical or different, each represent a monovalent hydrocarbon group selected from a linear or branched alkyl radical having 1 to 15 carbon atoms and a linear or branched alkoxyalkyl radical having 2 to 8 carbon atoms;
- the symbols R^2 and R^3, which are identical or different, each represent a monovalent hydrocarbon group selected from a linear or branched alkyl radical having 1 to 6 carbon atoms and a phenyl radical;
- A represents a removable group selected alternatively from: a halogen atom Hal belonging to chlorine, bromine and iodine atoms, the chlorine atom being preferred; or a radical para-$R^0-C_6H_4-SO_2-O-$ where R^0 is a linear or branched C1-C4 alkyl radical, the

tosylate radical para-CH_3-C_6H_4-SO_2-O- being preferred; or a radical R^0-SO_2-O- where R^0 is as defined above, the mesylate radical CH_3-SO_2-O- being preferred; or a radical R^0-CO-O- where R^0 is as defined above, the acetate radical CH_3-CO-O- being preferred, the most preferred radical A being the chlorine atom.

2. The process according to claim 1, characterized in that within the reactor a descending liquid fluid comprising the silane of formula (VII) and an ascending gaseous fluid comprising the alcohol of formula (VIII) will circulate in countercurrent.

3. The process according to claim 1 or 2, characterized in that the alcoholysis reaction is carried out within the reactor at a temperature between the boiling temperature of the alkanol of formula (VIII) and the boiling temperature of the starting silane of formula (VII), the reaction being carried out in the reactor alternatively at atmospheric pressure or at reduced pressure or at superatmospheric pressure.

4. The process according to claim 3, characterized in that the silane, 3-chloropropyldimethylchlorosilane, is introduced in the upper part of the reactor, the ethanol in the lower part, the reaction temperature in the column is greater than 77.80°C and less than 178°C at atmospheric pressure and the hydrochloric acid formed is stripped by the ethanol.

5. The process according to any one of claims 1 to 4, characterized in that the reaction is carried out in the presence of an organic solvent or an inert gas, said solvent having a boiling temperature at the operating pressure which is between the boiling temperature of the ethanol of formula (VIII) and that of the silane of the formula (VII).

6. The process according to claim 5, characterized in that the solvent is toluene, monochlorobenzene or xylene and the products corresponding to formulae (I) to (XI) have ethyl groups R^1 and methyl groups R^2 and R^3 and A and Hal represent a chlorine atom.

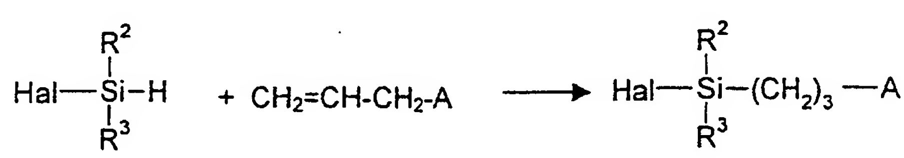
7. The process according to any one of claims 1 to 6, characterized in that the pressure inside the reactor is atmospheric pressure.

8. The process according to any one of claims 1 to 6, characterized in that the pressure inside the reactor is less than or greater than atmospheric pressure.

9. The process according to any one of claims 1 to 8, characterized in that the alcohol/silane molar ratio is greater than 1.

10. Process according to any one of claims 1 to 9, characterized in that the countercurrent reactor consists of a column equipped in its internal structure with a dumped or ordered packing or with plates.

11. A process for preparing the product of formula (VII) used as a starting reactant in the continuous process according to any one of claims 1 to 10 of the invention, characterized in that use is made of step (a), which proceeds according to the following equation:



(V) (VI) (VII)

where:

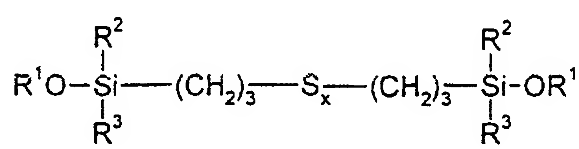
- the symbol Hal represents a halogen atom selected from chlorine, bromine and iodine atoms, the chlorine atom being preferred, and
- the symbols A, R^2 and R^3 are as defined above,

the reaction being carried out:

- by reacting, at a temperature ranging from -10°C to 200°C, one mole of the diorganohalosilane of formula (V) with a molar amount which is stoichiometric or different from the stoichiometry of the allyl derivative of formula (VI), the operation being carried out in a homogeneous or heterogeneous medium in the presence of an initiator consisting:
  - either of a catalytic activator consisting of: (i) at least one catalyst comprising at least one transition metal or one derivative of said metal, taken from the group consisting of Co, Ru, Rh, Pd, Ir and Pt; and optionally (2i) at least one hydrosilylation reaction promoter,
  - or of a photochemical activator, consisting in particular of appropriate ultraviolet radiation or appropriate ionizing radiation,

and optionally by isolating the diorganohalosilylpropyl derivative of formula (VII) that is formed.

12. A process for preparing bis(monoorganoxy-silylpropyl) polysulfides of formula:



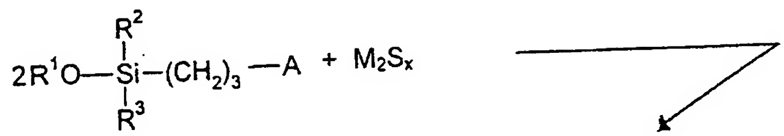
(I)

in which:

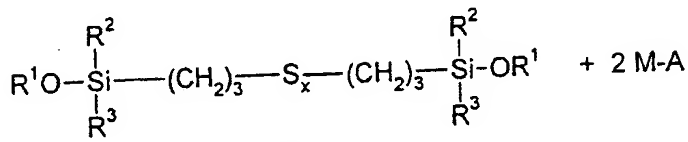
x is an integral or fractional number ranging from 1.5 ± 0.1 to 5 ± 0.1; and

the symbols R^1, R^2, R^3, Hal and A are as defined above,

by carrying out step (c), which proceeds according to the following equation:



(IX) (X)



(I)

where:
the symbols R^1, R^2, R^3, A and x are as defined above and the symbol M represents an alkali metal,
the reaction being carried out:
- by reacting, at a temperature ranging from 20°C to 120°C, either the reaction mixture obtained at the end of step (b) as defined in any one of claims 1 to 12, or the monoorganoxydiorganosilylpropyl derivative of formula (IX), taken in isolation after separation from said reaction mixture, with the metal polysulfide of formula (X) in the anhydrous state, using 0.5 ± 15 mol% of metal polysulfide of formula (X) per mole of the reactant of formula (IX) and optionally operating in the presence of an inert polar (or nonpolar) organic solvent, and
- by isolating the bis(monoorganoxysilylpropyl) polysulfide of formula (I) that is formed.

13. The process according to claims 11 and 12, characterized in that it is carried out by linking together steps (a), (b) and (c), in the definition of which the removable group A corresponds to the symbol Hal representing a halogen atom is a chlorine atom, and step (b) corresponds to the continuous process according to any one of the preceding claims 1 to 10.

14. The process according to claim 11 or 13, characterized in that step (a) is carried out by operating in the presence of a catalytic activator which comprises, as the catalyst(s) (i), one and/or other of the following metal species: (i-1) at least one finely divided elemental transition metal; and/or (i-2) a colloid of at least one transition metal; and/or (i-3) an oxide of at least one transition metal; and/or (i-4) a salt derived from at least one transition metal and a mineral or carboxylic acid; and/or (i-5) a complex of at least one transition metal equipped with organic ligand(s) which may possess one or more heteroatoms and/or organosilicon ligands; and/or (i-6) a salt as defined above in which the metal moiety is equipped with ligand(s) as also defined above; and/or (i-7) a metal species selected from the aforementioned species (elemental transition metal, oxide, salt, complex, complexed salt) where the transition metal is combined this time with at least one other metal selected from the class of the elements of groups 1b, 2b, 3a, 3b, 4a, 4b, 5a, 5b, 6b, 7b and 8 (with the exception of Co, Ru, Rh, Pd, Ir and Pt) of the Periodic Table (same reference), said other metal being taken in its elemental form or in a molecular form, it being possible for said combination to give rise to a bimetallic or polymetallic species; and/or (i-8) a metal species selected from the aforementioned species (elemental transition metal and transition metal/other metal combination; oxide, salt, complex and complexed salt on a transition metal base or on a transition metal/other metal combination base) which is supported on an inert solid support such as alumina, silica, carbon black, a clay, titanium oxide, an aluminosilicate, a mixture of aluminum and zirconium oxides, or a polymer resin.

15. The process according to claim 14, characterized in that step (a) is carried out by operating in the presence of a catalytic activator which comprises, as

the catalyst (or catalysts) (i), one and/or other of the metal species (i-1) to (i-8) where the transition metal belongs to the subgroup formed by Ir and Pt.

16. The process according to claim 15, characterized in that step (a) is carried out by operating in the presence of a catalytic activator which comprises, as the catalyst (or catalysts) (i), one and/or other of the metal species (i-1) to (i-8) where the transition metal is Ir.

17. The process according to claim 16, characterized in that step (a) is carried out by operating in the presence of a catalytic activator which comprises, as the catalyst (or catalysts) (i), at least one metal species of type (i-5) belonging to the iridium complexes of formula:

$$[Ir(R^4)Hal]_2 \quad (XI)$$

where:

- the symbol R^4 represents a conjugated or nonconjugated, linear or cyclic (mono- or polycyclic) polyene ligand having 4 to 22 carbon atoms and from 2 to 4 ethylenic double bonds;
- the symbol Hal is as defined above.

18. The process according to claim 12, characterized in that step (c) is carried out by deploying anhydrous metal polysulfides of formula (X) which are prepared beforehand from an alkali metal sulfide M_2S in the form of a hydrated sulfide, according to a procedure which consists in linking together the following operating phases (1) and (2):

- phase (1), where the alkali metal sulfide hydrate is dehydrated by applying the appropriate method which makes it possible to remove the water of crystallization while retaining the alkali metal

sulfide in the solid state throughout the dehydration phase;

- phase (2), where subsequently one mole of dehydrated alkali metal sulfide obtained is contacted with n(x-1) moles of elemental sulfur, the operating being carried out at a temperature ranging from 20°C to 120°C, optionally under pressure and optionally again in the presence of an anhydrous organic solvent, the aforementioned factor n being situated within the range from 0.8 to 1.2 and the symbol x being as defined above.

19. The process according to any one of claims 12 to 18, characterized in that the products corresponding to formulae (I), (V), (VI), (VII), (VIII) and (IX) have ethyl groups R^1 and methyl groups R^2 and R^3 and A and Hal represent a chlorine atom.